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I hereby Certify that this paper or fee is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service Under 37 CFR 1.10 on the date indicated above and is addressed to the Commissioner of Patents and Trademarks, Washington, D.C. 20231.

Name: Herbert M. Shapiro

Signature Herbert M. Shapiro

Case No.: IOS-118A

Hon. Commissioner of Patents and Trademarks  
 Washington, D.C. 20231

Sir:

Transmitted for filing is the Patent Application of

Inventor(s) :E. A. Mendoza Et Al.

Title :Thin Film Sol-Gel Derived Glass

Assignee :Intelligent Optical Systems, Inc. Torrance CA 90505

Priority(s) :

#### CLAIMS AS FILED AND AMENDED

FOR	NO. FILED	NO EXTRA	RATE	FEE
Total Claims	15	-	-	-
Indep. Claims	3	-	-	-
Mult. Dep. Claims	0	-	-	-
BASIC FEE				\$345.00
TOTAL FILING FEE				\$345.00

Enclosed are:

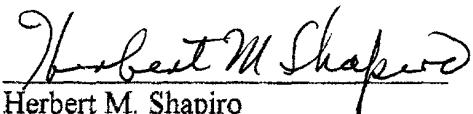
- \_\_\_\_ sheet(s) of formal drawings
- 2 sheet(s) of informal drawings
- 8 pages of specification with 15 claims and abstract
- in the English language
- in a non-English language
- Declaration and Power of Attorney
- An Assignment
- A certified copy of priority application(s)
- A preliminary Amendment reducing the number of claims for filing purposes

- A check to cover the filing fee
- A \$40.00 check for the assignment recording fee
- A \$ check for filing in a non-English language
- Declaration claiming Small Entity Status
- The right of priority under 35 USC 119 is claimed on the basis of the aforementioned foreign application(s)
- Applicant/Assignee claims Small Entity Status

Please send all correspondence to:

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Respectfully submitted,

  
Herbert M. Shapiro  
Herbert M. Shapiro  
Reg. No. 20578

Enclosures: [x]

**VERIFIED STATEMENT CLAIMING SMALL ENTITY STATUS (37 CFR 1.9(f)  
&1.27(c))—SMALL BUSINESS CONCERN**

Applicant or Patentee: **Edgar A. Mendoza**

Docket No. **IOS-118A**

Serial or Patent No.:

Filed or Issued: **Concurrently**

Title: **Thin Film Sol-gel Derived Glass**

I hereby declare that I am

- The owner of the small business concern identified below:  
 An official of the small business concern empowered to act on behalf of the concern identified below:

NAME OF SMALL BUSINESS CONCERN: **Intelligent Optical Systems, INC.**  
ADDRESS OF SMALL BUSINESS CONCERN: **2520 W. 237th Street, Torrance**

**CA 90505-5217**

I hereby declare that the above identified small business concern qualifies as a small business concern as defined in 37 CFR 1.9(d), for purposes of paying reduced fees to the United States Patent and Trademark Office, in that the number of employees of the concern, including those of its affiliates, does not exceed 500 persons. For purposes of this statement, (1) the number of employees of the business concern is the average over the previous fiscal year of the concern of the persons employed on a full-time, part-time or temporary basis during each of the pay periods of the fiscal year, and (2) concerns are affiliates of each other when either, directly or indirectly, one concern controls or has the power to control the other, or a third party of parties controls or has the power to control both.

I hereby declare that rights under contract or law have been conveyed to and remain with the small business concern identified above with regard to the invention described in:

- the specification filed herewith with title as listed above.  
 the application identified above  
 the patent identified above

If the rights held by the above identified small business concern are not exclusive, each individual, concern or organization having rights in the invention must file separate verified statements averring to their status as small entities, and no rights to the invention are held by any person, other than the inventor, who could not qualify as an independent inventor under 37 CFR 1.9(c) if that person made the invention, or by any concern which would not qualify as a small business concern under 37 CFR 1.9(d), or a nonprofit organization under 37 CFR 1.9(e).

Each person, concern or organization having any rights in the invention is listed below:

- no such person, concern, or organization exists.  
 each such person, concern or organization is listed below.

**Separate verified statements are required from each named person, concern or organization having rights to the invention averring to their status as small entities. (37 CFR 1.27).**

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I acknowledge the duty to file, in this application or patent, notification of any change in status resulting in loss of entitlement to small entity status prior to paying, or at the time of paying, the earliest of the issue fee or any maintenance fee due after the date on which status as a small entity is no longer appropriate. (37 CFR 1.28(b))

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application, any patent issuing thereon, or any patent to which this verified statement is directed.

NAME OF PERSON SIGNING Reuben Sandler  
TITLE OF PERSON IF OTHER THAN OWNER President & CEO  
ADDRESS OF PERSON SIGNING 2520 W. 237th Street, Torrance  
CA 90505-5217  
SIGNATURE Reuben Sandler DATE 5/18/00

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## **THIN FILM SOL-GEL DERIVED GLASS**

### **REFERENCE TO RELATED APPLICATIONS**

This application is related to a companion application Serial No. \_\_\_\_\_

(IOS-118), filed on even date herewith and assigned to the assignee of the present application.

### **FIELD OF THE INVENTION**

This invention relates to a process for producing photosensitive thin films of sol-gel derived glass and to such films of a thickness useful for integrated optic devices produced thereby..

### **BACKGROUND OF THE INVENTION**

The doctoral thesis by the applicant herein entitled "Photolithography of Integrated Optic Devices in Porous Glass, City University of New York, 1992 describes an organometallic system of inclusions in a thermally-assisted, porous glass bulk material. The process for fabricating the glass requires introduction of a photosensitizer, exposure to light through a mask and two heat treatments. The doctoral thesis states that sol-gel techniques can be used to make the porous glass bulk material.

### **BRIEF DESCRIPTION OF THE INVENTION**

The invention is based on the realization that the porous glass techniques for bulk materials using thermally assisted, organometallic, sol-gel derived glass can be extended to thin films suitable for telecom components and virtually free of lateral shrinkage. Consequently, a variety of highly desirable integrated optic components can be made by such a technique. Specifically, a technique for the photolithographic fabrication of integrated optic structures in thin films of photosensitive sol-gel glasses is described here.

This technique involves the formation of a photosensitive sol-gel thin film including an organometallic photosensitizer, on a suitable substrate (glass, silicon, or any other support material). Next, the photosensitive film is exposed to white or ultraviolet light inducing a photochemical reaction in the photosensitive sol-gel glass network with the end photo-product being a metal oxide. The photodeposited metal oxide is permanently bound to the sol-gel film glass network as a glass modifier during a heat treatment process, which in turn induces a permanent refractive index increase in the glass. The refractive index increase is dependent on the concentration of the photosensitizer and on the light energy used in the exposure process. Therefore, a spatially varying light intensity during exposure results in a spatially varying refractive index profile. This refractive index profile induced in the film can be designed to guide light.

Exposure of the photosensitive sol-gel film to white or ultraviolet light induces the unbinding of the metal from the photolabile moiety component of the photosensitizer followed by the binding of the metal to the sol-gel film. The exposed regions of the sol-gel film are converted to a metal oxide silica film by first and second step heatings at a low temperature and high temperature, respectively. The low temperature drives out the unexposed (unbound) photosensitizer and the unbound photolabile moiety. The higher temperature stepunbinds the organic component from the bound photosensitizer and drives it off. This step also permanently binds the metal to the silica film forming a metal oxide glass modifier. If the sol-gel film is deposited on a glass or silicon substrate, a metal oxide doped silica region of Si-O-M-O-Si is formed in the exposed regions acting as a glass modifier which in turn modifies the refractive index. The unexposed photosensitizer is driven off during the heat treatment steps. Since no material is

removed from the sol-gel film in this process, as in the case of prior-art processes, the resulting top surface is planar, thus leading to a simpler process for producing devices and for achieving increased lifetime of resulting devices.

## BRIEF DESCRIPTION OF THE DRAWINGS

Fig's. 1, 2, 4 and 5 are schematic side views of thin films in accordance with the principles of this invention; and fig. 3 is a block diagram of the steps for fabricating a structured thin film in accordance with this invention.

## DETAILED DESCRIPTION OF THE INVENTION

Fig. 1 is a side view of a film 11 of a sol-gel film with R-M-X constituents dissolved therein. The film is shown formed (usually by a well known spinning technique) on the SiO<sub>2</sub> surface layer 12 on a silicon substrate 13. The R constituents are taken from a class of volatile organic materials consisting of CH<sub>3</sub>, CH<sub>3</sub>-CH<sub>2</sub>, CH<sub>3</sub>-CH<sub>2</sub>-CH<sub>2</sub>, the M constituents (metals) are taken from the class of metals consisting of group IVA metals Ge, Sn and Pb, Group VI including Se and Te, Group VIII including Fe, Cu, Ni, and Group IVB including Ti and Zn and the X constituents (photosensitizers) are taken from the class consisting of chlorine, iodine, fluorine, bromine, and carbon.

Fig. 2 shows an alternate embodiment where the sol-gel film 20 is formed on a glass substrate 21. Fig's. 1 and 2 represent the initial sol-gel solution formed on appropriate substrates of silicon (fig. 1) and glass (fig. 2). The process of forming the sol-gel solution into useful film structures is discussed in connection with fig. 3.

Specifically, fig. 3 is a block diagram of the process for fabricating structured films from the sol-gel solution of fig's. 1 and 2. Block 31 of fig. 3 represents the step of

forming a sol-gel film with inclusions of R-M-X on a suitable substrate (as shown in fig. 1 or fig. 2). Block 32 represents the exposure of the film through a mask to light in a range of wavelengths from ultraviolet (UV) through the visible range. This step unbinds the photosensitizer (X) and binds the metal (M) to the silicon oxide.

Block 33 of fig. 3 represents the step of heating the film to about 300 degrees C for a time to bind the metal permanently to the SiO<sub>2</sub>. Block 34 of fig. 3 represents the final heating step to about 900 degrees C for driving off the metal and the photosensitizer from unexposed regions of the film.

Fig. 4 shows the structure of fig. 1 with a mask 40 in place. Mask 40 is opaque to the incident light (arrow 41) in regions 42 and 43 and is transparent to light in region 44. The result of exposure to light is a structured film (in excess of 1 micron) where the exposed region of the film includes Si – O – M – Si and the unexposed regions include SiO<sub>2</sub>.

The concentration of photodeposited metal oxide determines the index of refraction of the exposed region which can be made relatively high compared to that of adjacent regions. If we visualize region 44 extending away from the viewer as indicated by the broken lines in fig. 5, the resulting structure can be seen to represent a waveguide with the “core” being buried as indicated.

In one specific embodiment, a sol-gel film 1 - 10 microns thick was formed on a silicon substrate 1 cm x 0.5 cm x 0.1 cm thick with a SiO<sub>2</sub> surface layer < 2 microns thick thereon. The sol-gel film included Sn (M) 2% I (X) 2 %, and (CH<sub>3</sub>)<sub>3</sub> (R) 2%. Region 44 has a width of 10 microns, exposed to light with a wavelength of 254 nm for 30 minutes. The exposed region had an index of refraction of 1.55 and the unexposed

regions had indices of refraction of 1.45. The film has a thickness of 1 – 10 microns after processing and has unchanged lateral dimensions.

In another embodiment, a sol-gel film 1 – 10 microns thick was formed on a glass substrate 1 cm x 0.5 cm x 0.1 cm thick. The sol-gel film included Ti (M) 2% Cl (X) 4%, and Cp (R 4% where Cp is cyclopentadienyl. Region 44 has a width of 10 microns, exposed to light with a wavelength of 5.14 nm for 120 minutes. The exposed region had an index of refraction of 1.75 and the unexposed region had indices of refraction of 1.45. The film had a final thickness of 1 – 10 microns with the lateral dimensions thereof being unchanged.

## WHAT IS CLAIMED IS

1. A photosensitive thin film of thermally-assisted, organometallic, sol-gel derived glass doped with organometallic photosensitizer, including R-M-X inclusions where X is a photolabile moiety, M is a metal, and R is a volatile organic compound, said film having a thickness in excess of one micron.
2. A thin film as in claim 1, said film being formed on a substrate having a surface including silicon and oxygen.
3. A thin film as in claim 2 wherein said surface comprises SiO<sub>2</sub> and is a silica enriched thin layer on a silicon substrate.
4. A thin film as in claim 1, said film being formed on a glass substrate.
5. A thin film as in claim 1, said film where R is taken from a class of low-volatile organic molecules consisting of CH<sub>3</sub>, CH<sub>3</sub>-CH<sub>2</sub> and CH<sub>3</sub>-CH-2-CH<sub>2</sub> and (Cp), M is taken from a class consisting of metals in group IVA and IVB, transition metals and rare earth metals, and X is taken from a class consisting of and photolabile moiety including halogens and carbonyls.
6. A thin film as in claim 3 where R comprises CH<sub>2</sub>, M comprises Sn, and X comprises I.
7. A thin film as in claim 3 wherein R comprises cyclopentadienyl.
8. A thin film as in claim 3 wherein M comprises T.
9. A thin film as in claim 3 wherein X comprises Cl.
10. A thin film as in claim 4 wherein R comprises CH<sub>3</sub>.
11. A thin film as in claim 4 wherein M comprises Pb.
12. A thin film as in claim 4 wherein X comprises Cl.

13. A thin film as in claim 2 including thereon a mask opaque to light in the UV and visible ranges.

14. A thin film of sol-gel derived glass on a silica substrate, said film including at least one region of Si – O – M – O – Si with adjacent regions of SiO<sub>2</sub>, said film having a thickness substantially in excess of one micron and being free of cracks.

15. A method for forming a photosensitive sol-gel film including regions of different indices or refraction, said method comprising the steps of forming a photosensitive sol-gel film including an organometallic photosensitizer on a silica substrate, exposing said film through a mask to light of a wavelength and for a time for unbinding different amounts of metal constituents and of said sensitizers in different sections along at least a first channel thereof, exposing said film to heat at a first temperature and for a time to drive off the unbound sensitizers and to bind the metal constituents of said sol-gel film, and exposing said layer to heat at a second temperature higher than said first temperature for a time to unbind and drive off the organic constituents of said sol-gel film.

## ABSTRACT OF THE DISCLOSURE

Thermally-assisted organometallic sol-gel derived glasses have been found to permit fabrication of thin films sufficiently thin for telecom components. Inclusion of a photosensitizer in the film permits light of controlled intensity to modify refractive indices in the film to form useful structures.

Fig 1

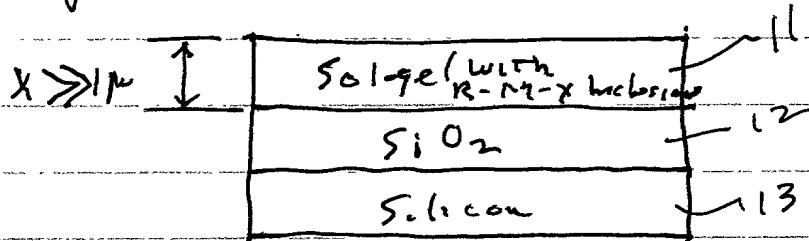


Fig 2

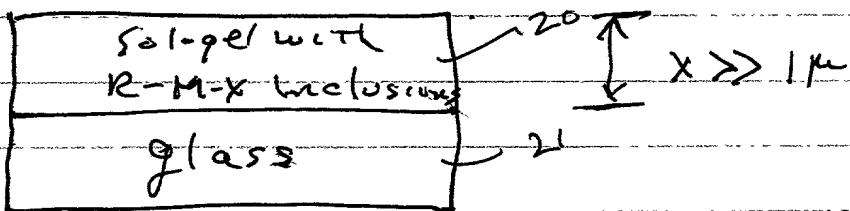


Fig 3

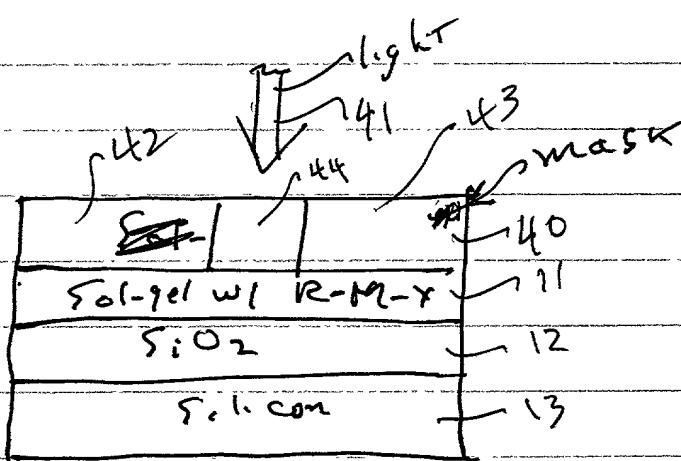


Fig 5

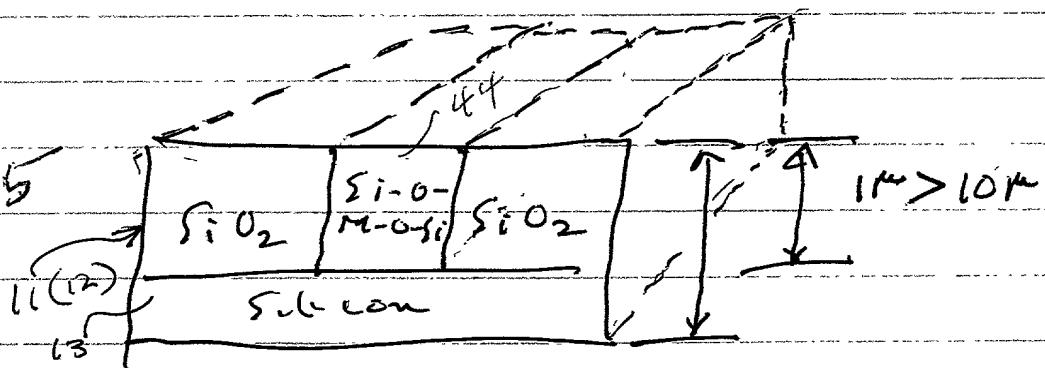
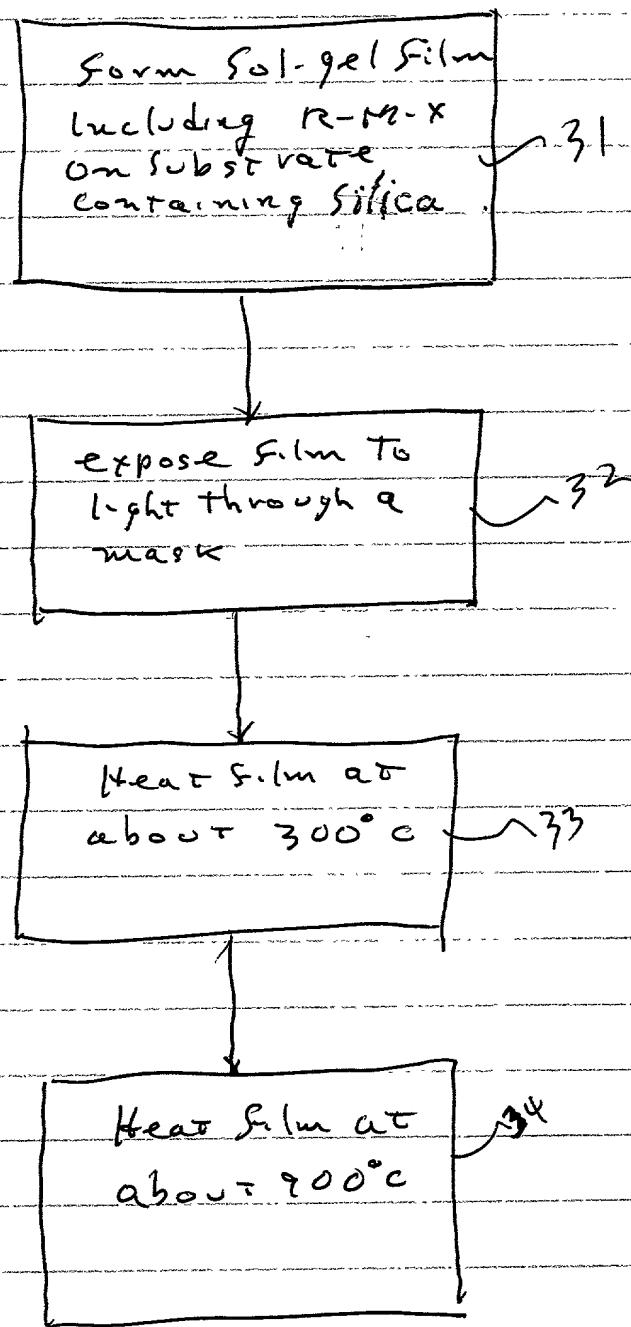


Fig 3



# DECLARATION AND POWER OF ATTORNEY FOR PATENT APPLICATION

## ENGLISH LANGUAGE DECLARATION

As below named inventor, I hereby declare that:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled

### **Thin Film Sol-Gel Derived Glass**

The specification of which

[X] is attached hereto

[ ] was filed on \_\_\_\_\_ as

application serial no. \_\_\_\_\_

and was amended on \_\_\_\_\_

(if applicable)

I hereby state that I have reviewed and understand the contents of the above identified specification, including claims as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations, Sec. L.56(a).

I hereby claim foreign priority benefits under Title 35, United States Code, Sec. 119 of any foreign application(s) for patent or inventors certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

Prior foreign application(s)	Priority claimed
------------------------------	------------------

(number)	(country)	(date/month/year/filed)	[ ] yes	[ ] no

I hereby claim the benefit under Title 35, United States Code, Sec. 120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner

provided by the first paragraph of Title 35, United States Code, Sec. 112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, Sec. 1.56(a) which occurred between the filing date of the prior application and the national or PCT filing date of this application:

(Application Serial No.)	(Filing Date)	status (patented pending, abandoned)
(Application Serial No.)	(Filing Date)	status (patented pending, abandoned)

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Sec. 1001 of Title 18, United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

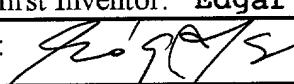
POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and transact all business in the Patent and Trademark Office connected therewith. (list name and registration number)

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Inventor's Signature:  Date: **5/18/00**

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Full name of second Inventor

Inventor's Signature Date

Residence

Citizenship

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